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PCT

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INTERNATIONAL APPLICATION PUBLISHED ACCORDING TO THE TREATY ON INTERNATIONAL COOPERATION IN THE FIELD OF PATENTS (PCT)

(51) International Patent classification: C 07 C 51/42, 51/44, 51/47, 59/08

(11) International publication No.: WO 98/55442 A1

(43) Int. Publication date: 10 Dec. 1998

(21) Int. file No. PCT/BE98/00080

(22) Int. Application date: 5, June 1998

(30) Priority dates: BE 9700489, 6 June 1997

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Published with international search report

(54) Title: **METHOD FOR PURIFYING LACTIC ACID**

(57) ABSTRACT: The invention concerns a method for recuperating and purifying a lactic acid solution obtained initially from a fermentation medium or any other source, consisting in a pre-treatment to eliminate the ion loads capable of catalyzing the lactic acid condensation and based on the principle of ion exchange or any other related principle; followed by at least one step of concentration at low temperature and brief retention time and at least one step of distilling the lactic acid using a thin layer process.

Lactic acid or 2-hydroxypropanoic acid is an α -hydroxylated carboxylic acid which may be produced by fermentation of various carbonated substrates either pure (glucose, saccharose, lactose,...) or impure (hydrolysis products of starch, molasses, whey etc.) with the aid of microorganisms such as bacteria of the genera *Lactobacillus*, *Pediococcus*, *Lactococcus* and *Streptococcus* or certain fungi such as *Rhizopus oryzae*. Other routes for the synthesis of lactic acid are known to the man of the art via chemical transformations of reagents of petrochemical origin such as hydrolysis of lactonitrile, the latter obtained also from acetaldehyde, the chlorination and hydrolysis of propionic acid or also by nitration of propane.

Lactic acid exists in two diastereo-isomeric forms: L(+) and D(-) lactic acid is encountered every day in new applications, ranging from the classical use as a food preservative up to the new developments such as the synthesis of solvents, pesticides, herbicides and biodegradable polymers. However, because

of the increasing strengthening of the required quality criteria and the necessity of achieving productions costs compatible with the market it is crucial to develop techniques for purification which are more effective and less costly.

Lactic acid may be purified either by precipitation in the form of metal lactates according to a neutralization reaction with sulfuric acid (Maesato K, Kamori A., Taki Chem. Co., JP 6 272 646 (25/09/85)) or by esterification with an alcohol, distillation and hydrolysis or the ester formed (Boroda, T.A., Polovko, V.N., Chistyakova, E.A., Pishch.Prom. 1966, 4, 35-8) or by electrodialysis (Jacquement, J.C., Rhone-Poulenc, DE 1 957 395 (14/11/68)). These products in the first case suffer from low quality of the product and high losses of lactic acid while in the other two cases the cost is prohibitive. A more recent process for purification consists in extracting lactic acid by liquid-liquid extraction by using at least one organic solvent not miscible with water either or not in the presence of at least one Lewis base such as a tertiary amine. According to this process the lactic acid should be recuperated in a second step by liquid-liquid extraction of the return flow. This step permits the lactic acid to be transferred back to water (Baniel, A.M., Blumberg, R., Hadju, K., IMI, DE 2 329 480 (19/06/72); Baniel, A.M., Miles Lab., EP 49 429 (06/10/80)). Finally lactic acid in the acid form and/or in the form of ammonium lactate or also metal lactate may be purified by passage through cationic and/or anionic ion exchange columns (Mapierala, W., Siminski, M., Przem. Ferment. Rolny. 1972, 16 (12), 4-10; Shkurino, O.V., Sauksha, V.E., Khim-Farm.Zh. 1986, 20(10), 1375-77; Maesato, K., Komori, A., Taki Chem. Co., JP 6 272 646 (25/09/85); Obara, H., Shimadzu Corp., JP 63 188 632 (30/01/87); Obara, H., Shimadzu Corp, JP 0 191 766 (30/09/87) Zeleneva, N.A., Ivanova, E.V., Karpushina, I.A., Gaevskaya, M.V., Teoriya i Praktika Sorbtsionnykh Protsessov, 1982, 67-69).

It is to be noted that all of the operations of purification are generally performed by starting with dilute solutions of lactic acid in water.

This is justified by the structure of the lactic acid which carries at the same time a hydroxyl function and a carboxylic acid group. In fact the

bifunctionality of lactic acid is the origin of the reactions of condensation which generate the lactoyl lactic, dilactoyl lactic, trilactoyl lactic, ... (n-lactoyl lactic) units which are also called the oligomers of lactic acid. These reactions of condensation or oligomerization tend toward an equilibrium but are more probable, the higher the concentration of the initial aqueous solution is (Holten, C.H.: "Lactic acid: Properties and chemistry of lactic acid and derivatives" Verlag Chemie, 1971). Figure 1 shows the equilibrium which exists between the monomeric form of lactic acid and its oligomers throughout the entire range of concentration in question.

The reactions of condensation or oligomerization of lactic acid corresponds in fact to the reactions of esterification; they are catalyzed by Brønsted and Lewis acids and bases. Therefore for the purpose of avoiding or minimizing intervention in these reactions it is imperative to eliminate all traces of impurities capable of catalyzing oligomerization by a preliminary purification step. In addition it is also recognized that temperature accelerates the formation of oligomers (Holten, C.H., "Lactic acid: Properties and chemistry of lactic acid and derivatives", Verlag Chemie, 1971). This explains why lactic acid in aqueous solution was long considered to be a substance of low volatility incapable of being distilled at 100°C. In fact lactic acid condenses to form oligomers whose boiling point is higher than 100°C at atmospheric pressure. From recent studies of the distillation of lactic acid by entrainment in steam at 160-200°C show that it is possible to distill it in yields of the order of 75-85%. Nevertheless these drastic conditions are prejudicial to quality of the products; degradation and racemization are impossible to avoid. A variant of distillation by steam entrainment is proposed by Noerdlinger (US Patent 924 494, DE 221 786 and DE 224 664). The technique consists of passing hot air or inert gas at high speed over the surface of a solution of lactic acid preliminarily freed of the water which it contains. The energy consumption and the low yield obtained, however, make this operation little viable industrially. For completeness we note that other apparatus and equipment profiles have been reported which

permit with more or less success the concentration and distillation of lactic acid in a dilute solution in water under reduced pressure in an evaporator displaying a very large evaporation surface relative to the volume of liquid involved (Sepitka, A., Prumysl Potravin 13, 385 and 605 (1962) and 14, 45 and 82 (1963); Shishkini, A.V., Domanskii, I.V., USSR, 709 613 (10/05/77)).

The present invention consists of a process for purification of lactic acid in aqueous solution such as is obtained in a fermentation medium or any other source preliminarily freed of solid compounds and/or biomass. As regards the step of separation of the solid compounds reference is made to the methods known to the art such as centrifugation, flocculation, microfiltration etc. On the other hand the process of purification described in this invention is original in the sense that it assures that one will obtain a very high quality of ^{lactic} ~~acetic~~ acid with a mass yield that is especially high and with minimal energy consumption. High quality means that the residual concentrations of mineral and organic impurities are such that the purified lactic acid may be utilized for pharmaceutical applications in conformity with all of the current pharmacopeias. Lactic acid purified by the process described in the present invention is more thermostable, i.e. it remains colorless after a heat treatment of 2 hours at 180°C and preserves the optical activity of the initial lactic acid used (stereospecific process). The term mass yield refers to the ratio expressed in per cent of the mass of the purified lactic acid to the mass of lactic acid initially used; these masses correspond to the lactic acid concentrations of 100%. It is well understood that applications requiring a lower purity may also be satisfied by the technique proposed in the present invention. The quantitative and selective aspects of this process of purification are assured by the joint use of (1) a pretreatment intended to eliminate substances capable of catalyzing the reaction of condensation of lactic acid, (2) conditions of temperature, holding time and viscosity permitting a reduction in intervention of the same reactions of condensation, and (3) conditions of temperature, holding time, viscosity and pressure and equipment profile making it possible to concentrate

up to a concentration by weight of 100%, and the distillation of the lactic acid.

Description of the Invention

The present invention describes a process for purification of lactic acid obtained from an aqueous solution of said acid such as is obtained in a fermentation medium or any other source previously freed of any solid substances and/or biomass possibly present. Figure 2 illustrates the process of purification of a lactic acid such as is dealt with by the present invention. This process consists essentially in the following steps:

1. Pretreatment of the dilute solution of the lactic acid (1)

The pretreatment considered within the scope of the invention consists of removal of ionic substances capable of catalyzing condensation or oligomerization of lactic acid. This pretreatment is performed at a low concentration of lactic acid, i.e. at a concentration lower than 80%, preferably lower than 50% and more preferably lower than 30%. A preferred approach to the present invention consists in removing the ionic substances by means of ion exchange resins. Therefore, by contacting the lactic acid solution with an anion exchange resin previously conditioned in the basic form (OH⁻) permits the replacement of the anionic impurities contained in the treated solution by hydroxide groups. The present invention is not restricted to the use of solid anion exchange resins but also includes any other technique known to the man of the art which permits the removal of the anionic charges to the benefit of the hydroxide ions such as the use of quaternated aliphatic amines present in the form of ammonium hydroxide in solution in at least one organic solvent not miscible with water. In this case the anion/hydroxide exchange takes place at the interface of the nonmiscible phases and is followed by a separation of the phases. A preferred variant of the invention consists in preceeding the step of anion exchange by a treatment which is characterized by the fact that the lactic acid solution is freed of cationic mono-, di-, tri- and/or multivalent charges possibly present. The cationic impurities are eliminated by contacting them with a cation exchange resin previously conditioned in an acid medium (H⁺). This variant is

preferred to the extent that it permits one to avoid the formation and precipitation of metal hydroxides which are poorly soluble in water during the anionic treatment. Here again the invention is not limited to cation exchange resins but also includes any other technique known to the art capable of exchanging the cations of the lactic acid solution for protons. For example, reference here is made to the use of a fatty acid of the carboxylic or sulfonic type in solution in at least one organic solvent which is not miscible with water. The cation/ proton exchange takes place at the interface between the nonmiscible solvents and is followed by a phase separation.

2. Concentration of the lactic acid solution (2)

This step of the invention consists in rapid concentration at low temperature of the lactic acid solution previously treated according to the method described as the first step of the invention (1) until a concentration between 50 and 90%, preferably between 70 and 90% is reached. A preferred variant of the present invention envisions conducting this evaporation under reduced pressure held between 50 and 500 mbar absolute and preferably between 50 and 250 mbar in order to assure a boiling point of the solution that is as low as possible. This step of the invention is performed by any technique known to the art such as evaporation of a trickling film.

3. Post-concentration of the lactic acid solution (3)

This step permits the post-concentration of the solution flowing out of the storage apparatus in step (2) up to a concentration of 100% lactic acid. The operation may advantageously be performed with a minimal holding time and temperature as low as possible in a thin film apparatus that is mechanically agitated (thin-film evaporator) or with the aid of a short path evaporator. The pressure is of the order of 10-500 mbar, preferably between 50 and 300 mbar and more preferably between 50 and 150 mbar. The temperature of the heated wall of the body of the evaporator is adjusted in such a way as to support the vaporization of the free water contained in the solution to be concentrated without overheating the same; the temperature is between 50 and 150°C, preferably between 80 and 120°C. Surprisingly it has been observed

that if lactic acid is present quantitatively in the form of the lactic acid monomer (and in the absence of free water, concentration = 100%) it is possible to distill it under reduced pressure in a reactor which maximizes the evaporation surface relative to the volume of the liquid. Besides claiming the use of such a reactor profile for distillation of the lactic acid the present invention guarantees the quantitative production of concentrated acid in monomer form which can be distilled for purification, properly speaking, by distillation.

4. Purification of lactic acid by distillation (4)

This step is characterized by the fact that the demineralized and concentrated lactic acid solution such as is produced in steps (1)-(3) is subjected to conditions such that the monomer (and to a smaller degree the dimer) of this acid distill quantitatively and selectively. The term quantitative here means that the totality of the fraction capable of distillation is effectively distilled. The term selective refers to the fact that only the monomer (and to a lesser degree the dimer) of the lactic acid distill(s) without entraining impurities or degradation products. This step is advantageously conducted in a reactor which maximizes the evaporation surface relative to the volume of the liquid, i.e. with a reactor which exploits the property of the thin film. A preferred variant of the present invention consists in utilizing for distillation of 100% lactic acid a mechanically agitated thin film evaporator on the outside of which the purified lactic acid is condensed (thin film evaporator) or a short path evaporator with an internal condenser. It is well known to the man of the art that such a system permits the heat exchange surface and the evaporation surface to be maximized. The temperature of the wall is held between 50 and 180°C, preferably between 80 and 160°C, more preferably between 110 and 160°C.

The pressure is between 10^{-3} and 10^{-2} mbar absolute, preferably between 10^{-1} and $2 \cdot 10^{-1}$ mbar absolute, more preferably between 1 and 10 mbar. A preferred variant of the present invention involves a vertical arrangement of the evaporator which permits a progression from a film under combined impulsion by

mechanical agitation and gravity. According to an improved but not essential variant of the present invention the residue of purification may be directed to a second distiller in which the conditions of temperature and pressure are more drastic (Figure 2, step 4 bis). The lactic acid coming from this post-distillation and partially purified may be recycled either to the feed of the primary distiller (step 4) or upstream from the process. A preferred version of the present invention involves the addition of a third body whose purpose is to facilitate the flow as a thin film and evaporation of the lactic acid during the step of distillation and/or post-distillation. This third body consists of any nontoxic substance which is chemically inert with respect to lactic acid, of low volatility, thermostable and of low viscosity under the conditions of distillation and post-distillation and preferably not miscible with lactic acid, in order to facilitate the separation by decantation and recycling. As an example we may mention the use of paraffins such as Fina Vestan A80B, A180B and preferably A360B has permitted drainage of the impurities and evaporation of the lactic acid all in accordance with the above mentioned requirements. Other details and specific features of the invention given in the following as nonlimiting examples stem from the description of several possible forms of its realization.

Examples

Example 1. Demineralization of a lactic acid solution

A solution of lactic acid obtained by fermentation is demineralized by percolation through solid ion exchange resins. The feed solution has the following analysis: lactic acid 185.1 g/l, pH 2.25, sulfate 1250 ppm, calcium 929 ppm, iron 15.8 ppm, potassium 133 ppm and sodium 98 ppm. This solution is fed at a rate of 3 BV/h to the top of a column containing 1 BV of macroporous strong cationic resin of the cross-linked polystyrene structure, Bayer Lewatit S 2528, previously conditioned in the H⁺ form by passing through 120 g of pure hydrochloric acid per liter of resin in the form of a 6% solution. The effluent recovered at the exit from this column is then directed to a column containing the same volume of anionic resin with an average basicity formed of ternary and quaternary amine groupings grafted onto a polystyrene structure

which is sold commercially by Bayer under the tradename of Lewatit S 432B. This resin is previously conditioned to the basic form by percolation of 120 g of pure caustic soda in the form of a solution of 4% concentration. Lactic acid solution treated under these conditions has the following average analysis after treatment of a volume corresponding to 15 times the volume of the cationic resins: lactic acid 167 g/l, pH 1.75, sulfates 0.7 ppm, calcium 0.8 ppm, iron 0.3 ppm, potassium 1.1 ppm and sodium 0.9 ppm. The penetration of the cationic resin which is manifested by an increase in the concentration of monovalent cations in the effluent from the first column of the system happens after the passage of 15 BV of the lactic acid solution. The penetration of the anionic resin demonstrated by the detection of sulfate ions in the effluent of the second column of the system takes place after treatment of 18 BV of feed solution.

Example 2. Concentration of a lactic acid solution up 80%.

A solution treated according to Example 1 is fed continuously into a trickling film evaporator of Inox displaying an evaporation surface of 0.31 m². The concentrated lactic acid solution is extracted at the same flow rate as the feed flow of the system (10.45 l/h) in order to maintain a constant level. The wall heating is assured by circulation of a heat-carrying oil in a double envelope. The conditions of pressure, temperature and the concentrations obtained are presented in Table 1 below.

Table 1. Concentration of an aqueous lactic acid solution 18.5 wt.% in an evaporator of the trickling film type of 0.31 m² evaporation surface.

Pressure (mbar)	Temp. (°C)	Desired concentration (wt.%)	Concentration ¹ obtained (wt.%)	Δ Concentration (wt.%)
98	46.9	70	68.3	-1.7
102	47.2	70	71.7	1.7
204	68.3	70	70.2	0.2
100	21.8	75	74.4	-0.6
103	56.4	80	79.6	-0.4
101	69	85	84.6	-0.4
197	82.6	85	86.5	1.5
96	68.3	85	82.1	-2.9

1) The concentration or total acidity is determined by acid-base titration after saponification.

Example 3. Post-concentration of lactic acid at various pressures.

An 81.75 wt.% lactic acid solution (degree of polymerization =13.19%) is fed continuously into a UIC mechanically stirred thin film borosilicate glass evaporator with an internal condenser (short path), having a heating surface and condensation surface of 0.06 m² preceded by a degassing preheater whose temperature is adjusted by the circulation of a heat-carrying oil. The assembly is held at a pressure of 50-250 mbar absolute. The results presented in Table 2 were obtained with a wall temperature of 100°C, a degassing temperature of 80°C, a condenser temperature of 15°C, a rotation speed on the rotor of 400 rpm and a feed flow rate of 1000 g/h.

Table 2. Post-concentration of an 81.75 wt.% lactic acid solution in a short path type evaporator UIC of 0.06 m².

Pressure (mbar)	Total acidity ¹ of concentrate (wt.%)	Total acidity ¹ of condensate (wt.%)	Degree ² of polymerization of concentrate (%)	Yield (%)
50	100.8	46.1	15.5	70.7
100	101.8	12.1	14.4	96.2
150	101.5	8.4	14.2	97.9
200	99.7	6.9	12.9	95.9
250	100.2	6.5	13.6	97.6

of the degree of polymerization as a function of holding time.

Table 3. Effect of heating time on the degree of polymerization

(1) The total acidity is determined by acid-basic titration after saponification.

(2) The degree of polymerization is defined as the ratio of the esterified acidity (wt.%) of carboxylic acid group in ester form to the total acidity.

Example 4. Effect of the heating time on the degree of polymerization (in the static system).

A solution of a demineralized lactic acid as obtained in Examples 1, 2 and 3 and having a concentration of 98.1 wt.% (degree of polymerization = 13.1%) is held at 100°C and atmospheric pressure for a variable time. Table 3 shows the dependence of a lactic acid solution with a total acidity of 98.1 wt.%.

Holding time (min)	Free acidity ¹ (wt.%)	Degree of polymerization ²
0	85.9	12.5
60	83.9	14.5
120	81.7	16.7
240	74.9	23.7
1050	65.0	33.8
1395	64.0	34.8

- (1) The free acidity is determined by acid-basic titration.
(2) The degree of polymerization is defined as the ratio of the esterified acidity to the total acidity or 98.1 wt.%.

Example 5. Effect of holding time on the degree of polymerization (in the dynamic system).

A solution of lactic acid with a concentration equal to 102 wt.% obtained in the same manner as described in the first three examples of the present invention is fed at a constant flow rate into a UIC mechanically agitated thin film borosilicate glass evaporator with an internal condenser (short-path) having a heating and condensation surface of 0.06 m². The system is held under a pressure of 40 mbar absolute, the temperature of the condenser and the temperature of the wall being respectively adjusted to 18 and 160°C (Table 4). Here it is assumed that all other parameters remain constant, the holding time in the apparatus in contact with the heated wall increases when the feed flow rate diminishes.

Table 4. Effect of temperature on the degree of polymerization of a solution of 102 wt.% lactic acid in a UIC 0.06 m² short path type evaporator.

Flow rate (g/h)	Total acidity (wt.%)	% n-mer ¹ (%)				
		Mono-	Di-	Tri-	Tetra-	Penta-
510	111.9	2.9	50.4	33.1	10.7	2.9
740	112.1	3.1	57.0	28.5	8.8	2.6
870	109.7	3.4	60.9	27.0	8.6	0.0

- (1) The percentage of n-mers is determined by volume exclusion chromatography (GPC).

Example 6. Effect of temperature on the degree of polymerization.

A solution of lactic acid with a concentration equal to 102 wt.% obtained in the same manner as described in the first three examples of the present invention is fed at a constant rate into a UIC mechanically agitated thin film borosilicate glass evaporator with an internal condenser (short path) having heating and condensation surfaces of 0.06 m². The system fed at a rate of 730 g/h is held under a pressure of 40 mbar absolute. The temperature of the condenser is maintained at 18°C (Table 5).

Table 5. Effect of temperature on the degree of polymerization of a short-path UIC 0.06 m² evaporator.

Temp. (°C)	Total acidity (wt.%)	% n-mer ¹ (%)				
		Mono-	Di-	Tri-	Tetra-	Penta-
140	108.5	17.4	52.3	21.7	8.5	0.0
150	107.6	7.1	62.8	21.9	6.1	2.4
160	109.3	3.1	57.0	28.5	8.8	2.6

(1) The percentage of n-mers is determined by volume exclusion chromatography (GPC).

Example 7. Distillation of lactic acid and effect of degree of polymerization on the distillation yield and quality of the distillate.

The solutions of lactic acids such as were obtained in Example 4 were introduced at a constant rate into a UIC mechanically agitated thin film borosilicate glass evaporator with an internal condenser (short path) having heating and condensation surfaces of 0.06 m², preceded by a degasser-preheater whose temperature is adjusted by circulation of a heat carrying oil. The assembly is held under a pressure of 5 mbar absolute. Table 6 summarizes the results obtained with a wall temperature of 140°C, a degassing temperature of 80°C and a condenser temperature of 15°C, a rotation speed of the rotor of 400 rpm and a feed rate between 798 and 915 g/h. The color of the distillate obtained is considered to be representative of its chemical purity.

Table 6. Effect of degree of polymerization on the yield of distillation and the quality of the distillate (short path UIC 0.06 m² evaporator).

Degree of polymerization of fluid involved (%)	Mass yield (%)	Total acidity of distillate (wt.%)	Degree of polymerization of distillate (%)	Color ¹ (Hazen)
14.5	89.8	96.5	4.1	0
	81.5	97.9	6.5	0
16.7	82.1	96.8	5.7	0
	76.4	97.0	6.6	0
23.7	66.3	96.2	7.6	20
	64.6	95.3	7.1	20
33.8	64.8	96.5	9.5	70
34.8	43.8	94.9	9.4	275

(1) The color is determined according to the APHA standard (American Public Health Association).

Example 8. Distillation of lactic acid and effect of ionic charge on the distillation yield.

A solution of demineralized lactic acid such as was obtained in Example 1, 2 and 3 and displaying a concentration of 101.46 wt.% is intentionally combined with sulfuric acid (98%). This solution is then introduced continuously into a UIC mechanically agitated thin film borosilicate glass evaporator with an internal condenser (short-path) displaying heating and condensation surfaces of 0.06 m², proceeded by a degasser-preheater whose temperature is adjusted by circulation of a heat carrying oil (Table 7). The assembly is held under a pressure of 3.5 mbar absolute. The conditions imposed on the system are the following: wall temperature 130°C, degassing temperature 84°C, condenser temperature: 10°C, rotation speed of rotor: 400 rpm.

Table 7. Effect of addition of sulfuric acid to a solution of lactic acid on the yield of distillation in a short path UIC 0.06 m² evaporator.

Content ¹ of exogenous protons (mmole/kg)	Content ¹ of exogenous sulfate ions (ppm)	Flow rate (g/h)	Mass yield (%)	Entrainment ¹ to degasser (wt.%)
0	0	622	65.2	14.2
		625	66.6	
		623	68.0	
2.1	100	630	64.6	15.6
		631	65.8	
		630	69.3	
5.2	200	626	66.7	14.6
		585	69.6	
8.3	400	623	68.0	
		618	68.5	
		622	69.6	
12.5	600	639	46.1	75.7
		640	45.3	
24.0	1150	644	43.6	
		647	37.8	

(1) By exogenous we refer to ions (protons and sulfates) introduced intentionally.

(2) The entrainment to the degasser signifies the percentage of the mass involved that is recovered from the degasser after bubble entrainment.

Example 9. Distillation of lactic acid and effect of temperature on the yield of distillation and the quality of the distillate.

A solution of lactic acid with a concentration equal to 98.12 wt.% obtained in the same manner as described in the first three examples of the present invention is fed at a constant flow rate into a UIC mechanically agitated thin film borosilicate glass evaporator with an internal condenser (short path) having heating and condensation surfaces of 0.06 m². The system fed at a rate of 870 g/h is held at a pressure of 5 mbar absolute. The temperature of the condenser is maintained at 50°C by circulation of water, and that of the degasser at 80°C by circulation of an heat carrying oil (Table 8).

Table 8. Effect of temperature on the yield of distillation and the quality of the distillate in an evaporator of the short path UIC 0.06 m² type.

Wall Temp. (°C)	Mass yield (%)	Color ¹ (Hazen)
130	7902	<5
140	82.7	<5
150	88.9	70

(1) The color was determined according to the APHA standard.

Example 10. Distillation of lactic acid and effect of holding time on quality of distillate.

The same lactic acid solution of 98.12 wt.% and the same experimental conditions as in Example 9 were repeated for two different flow rates respectively of 870 and 1120 g/h with a wall temperature of 150°C. As for Example 5, it is considered that the holding time in the apparatus is inversely proportional to the feed rate.

Table 9. Effect of feed rate on yield of distillation and quality of distillate in an evaporator of the short path
UIC 0.06 m² type.

Feed rate (g/h)	Mass yield (%)	Color ¹ (Hazen)
870	88.9	70
1120	85.5	20

(1) The color is determined according to the APHA standard.

Claims

1. Process for recovering and purification of an aqueous solution of lactic acid such as that obtained in a fermentation medium or other sources characterized by the fact that it consists of a pretreatment which eliminates the ionic substances capable of catalyzing the polycondensation of the lactic acid, at least one step of concentration which reduces the risk of polycondensation of the lactic acid and at least one step of selective and essentially quantitative distillation of the lactic acid.
2. Process as in claim 1 in which the concentration and the distillation of the lactic acid are performed according to the principle of the thin film.
3. Process according to claim 1 in which the distillation of the lactic acid makes use of a mechanically agitated thin film evaporator equipped with

an internal or external condenser.

4. Process as in any of claims 1-3 in which the distillation of the lactic acid is performed at a pressure between 10^{-3} and 10^{-2} mbar absolute and a heating surface temperature between 110 and 160°C.
5. Process as in any of claims 1-4 in which before distillation at least 1% and no more than 20% relative to its weight of an inert and immiscible compound is added to the lactic acid solution.
6. Process as in any of claims 1-5 in which the residue of distillation of the lactic acid is subjected to a second distillation and recycled.
7. Process according to claim 6 in which before distillation, to the residue of distillation at least 1% and no more than 20% relative to its weight of an inert and immiscible compound is added.
8. Process as in one or more of claims 1 and 2 in which the lactic acid is concentrated in two steps in order to achieve successively a concentration between 50 and 90 wt.% and a concentration of 100 wt.% of lactic acid under conditions of pressure between 50 and 500 mbar absolute.
9. Process according to claim 8 in which the second step of concentration is performed in a mechanically agitated thin film evaporator equipped with an internal or external condenser under a pressure between 10 and 500 mbar and at a temperature of the heating surfaces between 50 and 150°C.
10. Process as in any of claims 1-9 in which the pre-treatment is performed initially on an aqueous solution of lactic acid with a concentration less than 80 wt.%.
11. Process according to any of claims 1 and 10 in which the anionic and cationic impurities are eliminated with the aid of solid or liquid ion exchange resins.

International Search report in English appended.

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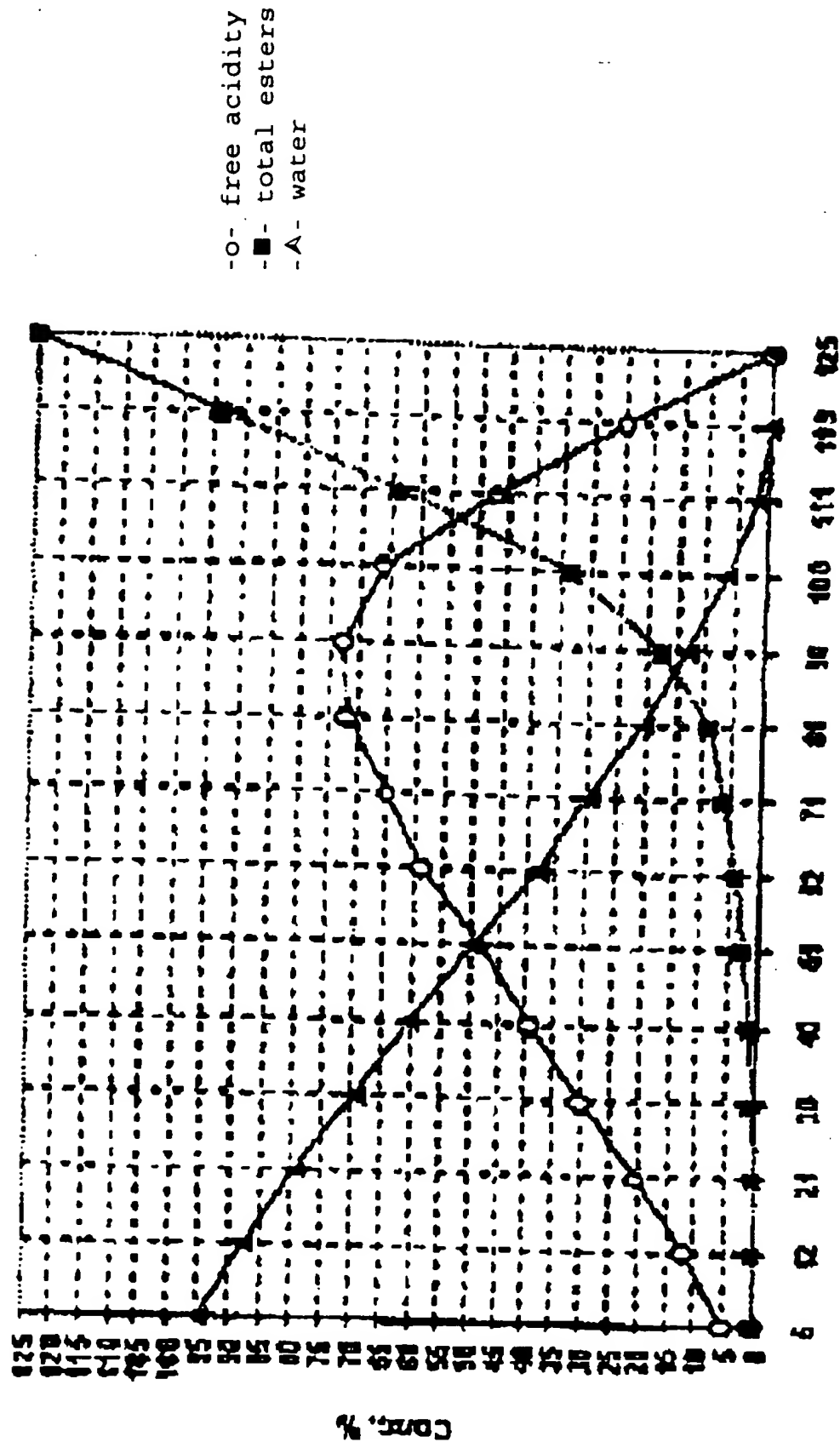


Figure 1. Dependence between total acidity and concentration of the aqueous lactic acid solution: (O) lactic acid monomer, (■) oligomers of the n-lactoyl lactic type and concentration in weight of water (Δ).

